



# PATENT COOPERATION TREATY

## PCT

### INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY

(Chapter II of the Patent Cooperation Treaty)

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference P66268	<b>FOR FURTHER ACTION</b> See Form PCT/IPEA/416	
International application No. PCT/EP2004/006700	International filing date (day/month/year) 21.06.2004	Priority date (day/month/year) 20.06.2003
International Patent Classification (IPC) or national classification and IPC C12P7/06, C12P5/02		
Applicant WILKENING, Carl Ludwing et al.		
<p>1. This report is the international preliminary examination report, established by this International Preliminary Examining Authority under Article 35 and transmitted to the applicant according to Article 36.</p> <p>2. This REPORT consists of a total of 5 sheets, including this cover sheet.</p> <p>3. This report is also accompanied by ANNEXES, comprising:</p> <p>a. <input checked="" type="checkbox"/> sent to the applicant and to the International Bureau) a total of 7 sheets, as follows:</p> <p style="margin-left: 40px;"><input checked="" type="checkbox"/> sheets of the description, claims and/or drawings which have been amended and are the basis of this report and/or sheets containing rectifications authorized by this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions).</p> <p style="margin-left: 40px;"><input type="checkbox"/> sheets which supersede earlier sheets, but which this Authority considers contain an amendment that goes beyond the disclosure in the international application as filed, as indicated in item 4 of Box No. I and the Supplemental Box.</p> <p>b. <input type="checkbox"/> (sent to the International Bureau only) a total of (indicate type and number of electronic carrier(s)) , containing a sequence listing and/or tables related thereto, in computer readable form only, as indicated in the Supplemental Box Relating to Sequence Listing (see Section 802 of the Administrative Instructions).</p>		
<p>4. This report contains indications relating to the following items:</p> <p><input checked="" type="checkbox"/> Box No. I Basis of the opinion</p> <p><input type="checkbox"/> Box No. II Priority</p> <p><input type="checkbox"/> Box No. III Non-establishment of opinion with regard to novelty, inventive step and industrial applicability</p> <p><input type="checkbox"/> Box No. IV Lack of unity of invention</p> <p><input checked="" type="checkbox"/> Box No. V Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement</p> <p><input type="checkbox"/> Box No. VI Certain documents cited</p> <p><input type="checkbox"/> Box No. VII Certain defects in the international application</p> <p><input type="checkbox"/> Box No. VIII Certain observations on the international application</p>		
Date of submission of the demand  10.02.2005	Date of completion of this report  21.09.2005	
Name and mailing address of the international preliminary examining authority:   European Patent Office - P.B. 5818 Patentlaan 2 NL-2280 HV Rijswijk - Pays Bas Tel. +31 70 340 - 2040 Tx: 31 651 epo nl Fax: +31 70 340 - 3016	Authorized Officer  Lejeune, R  Telephone No. +31 70 340-2347 	

# INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY

International application No.  
PCT/EP2004/006700

## Box No. I Basis of the report

1. With regard to the **language**, this report is based on the international application in the language in which it was filed, unless otherwise indicated under this item.
  - ☐ This report is based on translations from the original language into the following language, which is the language of a translation furnished for the purposes of:
    - ☐ international search (under Rules 12.3 and 23.1(b))
    - ☐ publication of the international application (under Rule 12.4)
    - ☐ international preliminary examination (under Rules 55.2 and/or 55.3)
2. With regard to the **elements\*** of the international application, this report is based on *(replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report):*

### Description, Pages

1-30 as originally filed

### Claims, Numbers

1-40 received on 10.02.2005 with letter of 04.02.2005

### Drawings, Sheets

1/3-3/3 as originally filed

- ☐ a sequence listing and/or any related table(s) - see Supplemental Box Relating to Sequence Listing

3. ☒ The amendments have resulted in the cancellation of:

- ☐ the description, pages
- ☒ the claims, Nos. 41-43
- ☐ the drawings, sheets/figs
- ☐ the sequence listing (*specify*):
- ☐ any table(s) related to sequence listing (*specify*):

4. ☐ This report has been established as if (some of) the amendments annexed to this report and listed below had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2(c)).

- ☐ the description, pages
- ☐ the claims, Nos.
- ☐ the drawings, sheets/figs
- ☐ the sequence listing (*specify*):
- ☐ any table(s) related to sequence listing (*specify*):

\* If item 4 applies, some or all of these sheets may be marked "superseded."

**INTERNATIONAL PRELIMINARY REPORT  
ON PATENTABILITY**

International application No.  
PCT/EP2004/006700

**Box No. V    Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement**

1. Statement			
Novelty (N)	Yes:	Claims	1-40
	No:	Claims	
Inventive step (IS)	Yes:	Claims	
	No:	Claims	1-40
Industrial applicability (IA)	Yes:	Claims	1-40
	No:	Claims	

2. Citations and explanations (Rule 70.7):

☐ see separate sheet

**Re Item V.**

The following document is referred to in this communication:

- D1 : BRYAN ET AL: "IOWA Ethanol Plant Pre-Feasibility"[Online] 2000, XP002300947 Retrieved from the Internet:  
URL:<http://www.iowaagopportunity.org/ethanolmanual/plant.pdf>; [retrieved on 2004-10-15]
- D2: DATABASE WPI Section Ch, Week 198151 Derwent Publications Ltd., London, GB; Class C03, AN 1981-95025D
- D3: DATABASE FSTA [Online] INTERNATIONAL FOOD INFORMATION SERVICE (IFIS), FRANKFURT-MAIN, DE; 1978, MARNICHENKO V A ET AL: "Intensified saccharification and fermentation of starchy raw material subjected to prior mechanical/chemical destruction." Database accession no. 79-2-11-h1802
- D4: US-A-5 559 031 (ZINNAMOSCA FRANCESCO ET AL) 24 September 1996 (1996-09-24)
- D5: BARUQUE FILHO EDMOND A ET AL: BIORESOURCE TECHNOLOGY, vol. 75, no. 1, October 2000 (2000-10), pages 49-55
- D6: US-B-6 355 4561 (HALLBERG DAVID E ET AL) 12 March 2002
- D7: WAYMAN M ET AL: STARCH, vol. 40, no. 11, 1988, pages 418-422
- D8: WEILAND P ET AL, WATER SCIENCE AND TECHNOLOGY, vol 22, no. 1-2, 1990, pages 385-394.
- D9: FUKUZAKI S ET AL: JOURNAL OF FERMENTATION AND BIOENGINEERING, vol 79, no. 4, 1995, pages 354-359

**Novelty (Art 33(2) PCT)**

The subject matter of claims 1-39 is new because the prior art does not disclose a method for producing ethanol and methane from biomass comprising obtaining methane from the "clear phase", where the particle size of less than 1 mm is explicitly mentioned.

The subject matter of claim 40 is new because although D1 discloses a production plant for producing ethanol and methane from biomass, D1 does not disclose a methane reactor

containing beads of 1 to 2 mm in which the methane bacteria are immobilized.

**Inventive step (Art 33(3) PCT)**

The subject matter of claims 1-40 does not involve an inventive step for the following reasons:

The production of both ethanol and methane from biomass are known (D1, D2).

D1 discloses a process for the production of ethanol from corn where a roller mill is used for grinding the corn (see page 38) and where the thin stillage (i.e. the supernatant after centrifugation of the stillage) is used as feedstock for the production of methane in an anaerobic digester (see page 43). The solids (which are defined in the present application as the particulate solids, i.e. not the dissolved solids, see page 6 of the application) are almost completely absent from the thin stillage (i.e. well below 1%).

D1 fails to disclose details concerning the particle size after milling and details concerning the methane reactor.

The prior art contains numerous disclosures where the influence of particle size in a process for the production of ethanol from biomass is discussed. These disclosures (e.g. D3, D4, D5 and D7) invariably direct the skilled person and also disclose the use of small (< 1 mm) particle sizes.

The methane reactors in the treatment of alcohol slops (stillage) are known in the prior art (see e.g. D8). Also high performance methane reactors are used (D9).

Consequently, the subject matter of claims 1-40 corresponds merely to the application to the different subprocesses of a bioethanol plant of the state of the art. There is no surprising or beneficial effect. Therefore, no inventive step can be acknowledged.

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February 2, 2005

Claims:

1. Method for producing ethanol and methane from biomass, comprising:
  - a) enzymatically liquefying and saccharifying flour of a biomass with a particle size of less than 1 mm in a conventional manner in the presence of water, thereby obtaining a mash;
  - b) fermenting and distilling the substrate in a conventional manner, thereby obtaining ethanol and a pulp;
  - c) separating the pulp into a solid phase and a clear phase, wherein a clear phase with a content of solids of less than 1% is obtained;
  - d) obtaining methane from the clear phase in a high-performance methane reactor.
2. Method according to claim 1, comprising milling biomass to a particle size of less than 1 mm, thereby producing flour.
3. Method according to any of the preceding claims, wherein hull components are substantially separated from the flour prior to step a, or separated from the mash prior to step b.
4. Method according to any of the preceding claims, wherein the biomass is grain.
5. Method according to any of the preceding claims, wherein grain, in particular wheat, rye, maize or triticales is used as biomass, and the bran is separated after milling.
6. Method according to any of the preceding claims, wherein the particle size of the flour is less than 0,6 mm.



7. Method according to any of the preceding claims, wherein proteins present in the biomass are substantially separated from the flour prior to step a or separated from the mash prior to step b or separated from the clear phase of the pulp in step c.
8. Method according to claim 7, wherein the separation of the proteins prior to step b comprises precipitation by cooling and separation of the precipitate.
9. Method according to claim 7, wherein the separation of the proteins in step c comprises precipitation by cooling and separation of the precipitate.
10. Method according to claim 9, wherein yeast, fibres, solid substances, fat and/or proteins present in the pulp are agglomerated by cooling and sedimented prior to separation of the pulp into solid phase and clear phase.
11. Method for producing ethanol und methane from grain, comprising
  - a) milling the grain to a particle size of less than 1 mm and separating the bran from the flour;
  - b) enzymatically liquefying and saccharifying the flour in a conventional manner in the presence of water, thereby obtaining a mash;
  - c) substantially precipitating the proteins present in the mash by cooling, sieving and drying, thereby obtaining the proteins and a substrate;
  - d) fermenting and distilling the substrate in a conventional manner, thereby obtaining ethanol and pulp;
  - e) separating the pulp into a solid phase and a clear

phase, wherein a clear phase with a content of solids of less than 1% is obtained; and

- f) obtaining methane from the clear phase in a high-performance methane reactor.

12. Method according to any of the preceding claims, wherein a decanter or a disk centrifuge is used for separation of the solid phase and clear phase of the pulp.

13. Method according to any of the preceding claims, wherein about 80% of the liquid in the pulp is withdrawn with the clear phase.

14. Method according to any of the preceding claims, wherein the content of solids in the clear phase is less than 0,5%.

15. Method according to any of the preceding claims, wherein fermentation is carried out in a batch process, cascading process or in a continuous process comprising a recycling of yeast.

16. Method for producing ethanol and methane from grain, comprising

- a) milling the grain to a particle size of less than 1 mm, preferably less than 0,6 mm, and separating bran and hull components from the flour;

- b) enzymatically liquefying and saccharifying the flour in a conventional manner in the presence of water, thereby obtaining a mash;

- c) fermenting and distilling the substrate in a conventional manner, thereby obtaining ethanol and pulp;

- d) agglomerating yeast, fibres, solid substances, fat and/or proteins present in the pulp by cooling and sedimenting them;



- e) dividing the pulp into a solid phase and a clear phase, wherein a clear phase with a content of solids of less than 1% is obtained; and
  - f) obtaining methane from the clear phase in a high-performance methane reactor.
17. Method according to any of the preceding claims, wherein a high-performance methane reactor is employed, comprising beads with a diameter of 1 to 2 mm in which methane bacteria are immobilised.
18. Method according to claim 17, wherein the immobilisation of the methane bacteria in the beads increases the space time yield in the reactor and preferably allows a space time yield of at least 25 kg CSB/(m<sup>3</sup>\*d).
19. Method according to any of the preceding claims, wherein the methane production in a high-performance methane reactor comprises a pre-acidification / conditioning.
20. Method according to any of the preceding claims, wherein the high-performance methane reactor comprises an Upflow anaerobic sludge blanket (UASB)-reactor.
21. Method according to any of the preceding claims, wherein the high-performance methane reactor comprises an Internal Circulation (IC)-reactor.
22. Method according to any of the preceding claims, wherein the crude ethanol is rectified and, if necessary, dehydrated, in order to obtain bioethanol or neutral ethanol.
23. Method according to any of the preceding claims, wherein more than 100 m<sup>3</sup> Ethanol / day are produced.

24. Method according to any of the preceding claims, wherein more than 300 m<sup>3</sup> Ethanol / day are produced.
25. Method according to any of the preceding claims, wherein the clear phase of the pulp is aerobically purified after anaerobic purification in the methane reactor.
26. Method according to claim 25, wherein the anaerobically/aerobically purified clear phase is added to the conversion process as water for dilution.
27. Method according to any of claims 25 and 26, wherein anaerobically/aerobically purified clear phase is employed for the addition of water for liquefaction of the flour.
28. Method according to any of the preceding claims, wherein the solid phase of the pulp is mixed with separated hull components and/or bran.
29. Method according to any of the preceding claims, wherein the solid phase of the pulp is mixed with separated proteins.
30. Method according to claims 28 or 29, wherein the mixture is further dried.
31. Method for producing a feeding stuff and/or fertilizer comprising a method according to claims 28 to 30.
32. Method for producing energy and/or heat, comprising a method for producing ethanol and methane according to any of claims 1 to 31 and converting the methane to energy and/or heat.
33. Method according to claim 32, wherein the solid phase of the pulp is dried and burned for the generation of energy.
34. Method for producing energy and/or heat, comprising a method for producing ethanol and methane from grain, comprising

- a) milling the grain to a particle size of less than 0,6 mm and separating bran and hull components from the flour;
  - b) enzymatically liquefying and saccharifying the flour in a conventional manner in the presence of water, thereby obtaining a mash;
  - c) fermenting and distilling the substrate in a conventional manner thereby obtaining ethanol and pulp;
  - d) agglomerating yeast, fibers, solid substances, fat and/or proteins by cooling and sedimenting them;
  - e) dividing the pulp into a solid phase and a clear phase, wherein a clear phase with a content of solids of less than 1% is obtained; and
  - f) obtaining methane from the clear phase in a high-performance methane reactor and drying and burning the solid phase of the pulp for the generation of energy.
35. Use of the clear phase of pulp from the production of bioethanol with a content of solids of less than 1 % (w/v) for producing methane, energy and heat.  
wherein a high-performance methane reactor is employed for production of methane, comprising beads with a diameter of 1 to 2 mm in which methane bacteria are immobilised.
36. Use according to claim 35, wherein the immobilisation of the methane bacteria in the beads increases the space time yield in the reactor and preferably allows a space time yield of at least 25 kg CSB/(m<sup>3</sup>\*d).
37. Use according to any of claims 35 or 36, wherein the method of preparing methane in a high-performance methane reactor comprises a pre-acidification / conditioning.

38. Use according to any of claims 35 to 37, wherein the high-performance methane reactor comprises an Upflow anaerobic sludge blanket (UASB) reactor.

39. Use according to any of claims 35 to 38, wherein the high-performance methane reactor comprises an Internal Circulation (IC) reactor.

40. Production plant for producing ethanol and methane from a biomass in accordance with any of claims 1 to 34 comprising a means for fermentation, distillation, and a high-performance methane reactor.

(C) WPI / DERWENT

AN - 1981-95025D [51]  
CPY - PROC-N  
DC - C03 D13 D16 E17 E36  
DR - 0245-P 0323-P  
FS - CPI  
IC - C12C0/00  
IN - MULLER H  
MC - C04-B04A C10-E04D C10-J02 D03-F01 D05-B D05-C E10-E04E  
M1 - [03] M423 M720 M903 N132 N134 N153 N164 Q211 Q212 Q213 Q214 Q232 Q241  
V500 V550 V752  
M2 - [01] H4 H401 H481 H8 M210 M212 M272 M281 M320 M416 M620 M720 M903 M910  
N132 N134 N153 N164 Q232 Q241  
- [02] M210 M211 M320 M416 M610 M620 M720 M903 M910 N132 N134 N153 N164  
Q232 Q241  
M3 - [01] H4 H401 H481 H8 M210 M212 M272 M281 M320 M416 M620 M720 M903 M910  
N132 N134 N153 N164 Q232 Q241  
- [02] M210 M211 M320 M416 M610 M620 M720 M903 M910 N132 N134 N153 N164  
Q232 Q241  
PA - (PROC-N) PROCESS ENG CO  
PN - ZA8005297 A 19810710 DW198151 016pp  
PR - ZA19800005297 19800827  
XIC - C12C-000/00  
AB - ZA8005297 Prodn. of alcohol from corn or other types of grain  
comprises (a) degerminating the grain to separate a starch fraction,  
(b) subjecting this fraction to two-stage saccharification to give a  
sugar mash, removing insoluble protein from the mash and fermenting  
the mash to produce alcohol, and (c) removing the alcohol by distn.  
and subjecting the residue to anaerobic decomposition to produce CH4  
gas.  
- The insoluble protein is recovered for human consumption and in the  
overall process less heat energy is required than in prior processes.  
IW - PRODUCE ALCOHOL CORN DE GERMINATE TWO-STAGE SACCHARIFICATION PROTEIN  
REMOVE FERMENTATION  
IKW - PRODUCE ALCOHOL CORN DE GERMINATE TWO-STAGE SACCHARIFICATION PROTEIN  
REMOVE FERMENTATION  
INW - MULLER H  
NC - 001  
OPD - 1980-08-27  
ORD - 1981-07-10  
PAW - (PROC-N) PROCESS ENG CO  
TI - Prodn. of alcohol from corn - by de-germination, then two=stage  
saccharification, protein removal and fermentation